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Letters to the Editor

On the structure of hydroxylamine uranium (IV) flouride

By T. Ratho, T. Patel and B. Sahu*

Department of Physics, Regional Engineering College, Rourkela, India,

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Hydroxylamine uranium (IV) flouride (UF_4. NH_2. OH. HF) is obtainable in the form of micro-crystals, green in colour. As the substance did not show the possibility of yielding any suitable single crystal, the powder method of study was undertaken. In our previous paper (Ratho *et al* 1968), we have found out the unit cell dimensions, number of molecules per unit cell and space group of hydrazine uranium (IV) flouride ($N_2H_5UF_5$). Such study will throw light on how the different groups are rearranging themselves with uranium.

The chemically pure substance was taken and filtered CuK_α (λ=1.54Å) radiation, obtained from a Machlett A-2 X-ray Diffraction tube operated at 30 KV and 20 mA, was used to irradiate the sample in a 9 cm diameter Rigaku camera for 16 hours to record a suitable powder pattern. The rings on the photograph were measured and the corresponding $\sin^2\theta$ values and inter-planar spacings d were recorded in the table. Failing to fit the data to cubic, tetragonal and hexagonal systems the Lipson's (Lipson 1949) method was worked out. The difference diagram and the appearance of good number of constant differences forecasted that the crystal may be orthorhombic.

For orthorhombic crystal, we know,

$$\sin^2\theta = Ah^2 + Bk^2 + Cl^2$$

where $A = \lambda^2/4a^2$, $B = \lambda^2/4b^2$ and $C = \lambda^2/4c^2$.

* Dept. of applied chemistry, I.I.T., Kharagpur.

TABLE 1.

Spacings 'd' and intensity	$\sin^2\theta$ obs.	$\sin^2\theta$ cal.	Indices	Spacings 'd' and intensity	$\sin^2\theta$ obs.	$\sin^2\theta$ cal.	Indices
3.65420 vvw	0.04442	0.04446	300	1.51651 vvw	0.25785	0.25779	292
		0.04379	230			0.25792	084
						0.25818	721
3.46227 m	0.04946	0.04990	301			0.25832	246
		0.04896	003			0.25776	445
3.27860 m	0.05516	0.05514	320	1.48351 vvw	0.26937	0.26952	643
3.00434 w	0.06569	0.06622	302	1.43830 vvw	0.28658	0.28676	2,10,0
		0.06555	232			0.28659	275
						0.28632	207
2.84559 vvw	0.07322	0.07299	033				
		0.07393	331	1.38922 vw	0.30732	0.30688	085
						0.30760	644
2.60890 vvw	0.08710	0.08704	004			0.30666	564
		0.08715	411			0.30705	356
		0.08651	250				
		0.08718	340	1.37573 vvw	0.31326	0.31369	317
2.34326 vvw	0.10797	0.10827	252	1.35443 vw	0.32218	0.32160	801
		0.10851	431			0.32170	327
						0.32201	516
2.12810 m	0.13096	0.13083	070				
		0.13161	511	1.24103 vw	0.38497	0.38448	0,12,0
		0.13150	304			0.38436	626
		0.13067	413	1.22243 vvw	0.39577	0.39594	5,10,1
2.05994 w	0.13973	0.13962	521			0.39529	318
						0.39585	754
2.02960 vvw	0.14391	0.14361	115				
		0.14352	442	1.16791 vw	0.43473	0.43438	3,12,1
						0.43404	862
1.84500 w	0.17417	0.17355	254			0 43470	782
		0.17422	344				
				1.07350 vvw	0.51454	0.51450	7,10,1
1.82424 m	0.17817	0.17784	600			0.51467	816
		0.17812	045			0.51438	548
1.74780 vvw	0.19429	0.19396	621	1.02760 vw	0.56210	0.56232	6,12,0
		0.19475	453			0.56222	449
						0.56187	497
1.66852 vvw	0.21297	0.21240	282				
		0.21321	514				
1.56420 vvw	0.24223	0.24206	700				
		0.24232	642				

With the help of the above equation all the lines on the powder pattern could be easily indexed, and the constants obtained are $A = 0.00494 \pm 0.00006$, $B = 0.00267 \pm 0.000035$ and $C = 0.005444 \pm 0.000065$. Thus the cell dimensions obtained are $a = 10.9630 \pm 0.0660$ Å, $b = 14.9024$Å ± 0.0890Å and $c = 10.4391 \pm 0.0630$Å.

The experimental value of the density of the crystal was found out to be 4.3501 gm/cc and the calculated density is 4.2909 gm/cc. The number of molecules per unit cell comes out to be 12. The study of indices shows, hkl, hk0, h0l, 0kl, h00, 0k0, and 00l to have no condition. Therefore the probable space group assigned to the crystal is P222 or Pmm2 or Pmmm.

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On the structure of dithiocyanato-tetrakis γ-picoline zinc (II)

By T. Ratho and T. Patel

Department of Physics, Regional Engineering College, Rourkela, India

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Dithiocyanato-tetrakis γ—picoline zinc (II) $[Zn(\gamma-C_6H_7N)_4(CNS)_2]$ is white in colour and obtainable in the form of microcrystals. As it was not possible to develop single crystals out of it, the powder method of analysis was followed for identification. The Debye-Scherrer powder pattern was obtained in six hours by a 9 cm diameter Rigaku camera using filtered CuK_α radiation obtained from a Machlett A-2 X-ray diffraction tube running at 30 KV and 15 mA.

Measuring the line positions on the film, Q_{hkl} values for the lines were computed accurately (table 1). A systematic analysis of the data after Azaroff *et al* (1958) and Henry *et al* (1951) eliminated the possibility for the crystal belonging to any higher symmetry.